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Form Approved OMB NO. 0704-0188

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1. REPORT DATE (DD-MM-YYYY)	2. REPORT TYPE	3. DATES COVERED (From - To)
28-11-2012	Final Report	1-Sep-2009 - 31-Aug-2012

4. TITLE AND SUBTITLE	5a. CONTRACT NUMBER
A Fundamental Investigation of Infrared Properties of Graphene for Potential Sensor Applications	W911NF-09-1-0437
	5b. GRANT NUMBER
	5c. PROGRAM ELEMENT NUMBER
	611102

6. AUTHORS	5d. PROJECT NUMBER
Elias Towe	
	5e. TASK NUMBER
	5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAMES AND ADDRESSES	8. PERFORMING ORGANIZATION REPORT NUMBER
Carnegie Mellon University Office of Sponsored Programs Carnegie Mellon University Pittsburgh, PA 15213 -	

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)	10. SPONSOR/MONITOR'S ACRONYM(S) ARO
U.S. Army Research Office P.O. Box 12211 Research Triangle Park, NC 27709-2211	11. SPONSOR/MONITOR'S REPORT NUMBER(S) 56231-EL.3

12. DISTRIBUTION AVAILIBILITY STATEMENT
Approved for Public Release; Distribution Unlimited

13. SUPPLEMENTARY NOTES
The views, opinions and/or findings contained in this report are those of the author(s) and should not contrued as an official Department of the Army position, policy or decision, unless so designated by other documentation.

14. ABSTRACT
This document is a final report for a project on graphene performed at Carnegie Mellon from September 1, 2009 – August 31, 2012. The project goals were twofold: (i) construct a growth system for synthesizing high quality, and (ii) investigate the properties of the material for potential infrared sensing. The first goal has been successfully met; the second has yielded results that have been informative on future directions on the fabrication of graphene-based infrared sensors. We have determined, for example, that it is possible to dope graphene p-type and

15. SUBJECT TERMS
Graphene; Two-Dimensional Material; Infrared; Silicon Carbide; CVD

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	15. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT	b. ABSTRACT	c. THIS PAGE			Elias Towe
UU	UU	UU	UU		
					19b. TELEPHONE NUMBER
					412-268-8091

Standard Form 298 (Rev 8/98)
Prescribed by ANSI Std. Z39.18

Report Title

A Fundamental Investigation of Infrared Properties of Graphene for Potential Sensor Applications

ABSTRACT

This document is a final report for a project on graphene performed at Carnegie Mellon from September 1, 2009 – August 31, 2012. The project goals were twofold: (i) construct a growth system for synthesizing high quality, and (ii) investigate the properties of the material for potential infrared sensing. The first goal has been successfully met; the second has yielded results that have been informative on future directions on the fabrication of graphene-based infrared sensors. We have determined, for example, that it is possible to dope graphene p-type and n-type with metals of appropriate work functions relative to graphene. Doping is essential for the formation of p-n junctions that can be used in light detection. Furthermore, the results of the project suggest that it may be advantageous to embed graphene in an optical cavity to enhance its absorbance so that sufficient photocurrent can be generated when the material is illuminated. This requirement is in view of the single atomic- or bi-layer nature of graphene samples that are likely to be used in active media of detector structures.

Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

Received	Paper
08/31/2011	1.00 Ashwin Ramasubramaniam, Doron Naveh, Elias Towe. Tunable Band Gaps in Bilayer Graphene?BN Heterostructures, Nano Letters, (03 2011): 1070. doi: 10.1021/nl1039499
11/28/2012	2.00 Doron Naveh, Elias Towe, Ashwin Ramasubramaniam. Tunable band gaps in bilayer transition-metal dichalcogenides, Physical Review B, (11 2011): 0. doi: 10.1103/PhysRevB.84.205325
TOTAL:	**2**

Number of Papers published in peer-reviewed journals:

(b) Papers published in non-peer-reviewed journals (N/A for none)

Received	Paper
TOTAL:	

Number of Papers published in non peer-reviewed journals:

(c) Presentations

1. A. Ramasubramaniam D. Naveh, and E. Towe, "Tunable band gaps in bilayer graphene-BN heterostructures," Bulletin of the American Physical Society, APS March Meeting 2011 Vol 56(1) Paper ID: BAPS.2011.MAR.H37.5 (2011).

2. D. Naveh, A. Ramasubramaniam, and E. Towe "Electronic and transport properties of few-layer MoS2 Crystals," Bulletin of the American Physical Society, APS March Meeting 2012 Vol. 57(1), Paper ID: BAPS.2012.MAR.Z17.1 (2012).

3. A. Ramasubramaniam D. Naveh, and E. Towe, "Tunable band gaps in transition metal dichalcogenides," Bulletin of the American Physical Society, APS March Meeting 2012 Vol. 57(1), Paper ID: BAPS.2012.MAR.V7.10 (2012).

Number of Presentations: 3.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received   Paper

TOTAL:

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received   Paper

TOTAL:

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):

(d) Manuscripts

Received   Paper

TOTAL:

Number of Manuscripts:

Books

TOTAL:

Patents Submitted

Patents Awarded

Awards

Graduate Students

NAME	PERCENT_SUPPORTED	Discipline
Ellen Reifler	0.20	
FTE Equivalent:	**0.20**	
Total Number:	**1**	

Names of Post Doctorates

NAME	PERCENT_SUPPORTED
Doron Naveh	0.80
Ibrahim Kimukin	0.00
FTE Equivalent:	**0.80**
Total Number:	**2**

Names of Faculty Supported

NAME	PERCENT_SUPPORTED	National Academy Member
Elias Towe	0.08	
FTE Equivalent:	**0.08**	
Total Number:	**1**	

Names of Under Graduate students supported

NAME	PERCENT_SUPPORTED	Discipline
Madeleien MCKay	0.00	Physics
Elias Szabo-Wexler	0.00	electrical and Computer Engineering
FTE Equivalent:	**0.00**	
Total Number:	**2**	

Student Metrics
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The number of undergraduates funded by your agreement who graduated during this period and will receive
scholarships or fellowships for further studies in science, mathematics, engineering or technology fields: 0.00

Names of Personnel receiving masters degrees

NAME

Total Number:

Names of personnel receiving PHDs

NAME

Total Number:

Names of other research staff

NAME	PERCENT_SUPPORTED

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Sub Contractors (DD882)

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Scientific Progress

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Technology Transfer

A Fundamental Investigation of Infrared Properties of Graphene for Potential Sensor Applications

E. Towe (PI)

Performance Period: September 1, 2009 – August 31, 2012

Date of Report: November 27, 2012

Submitted to

Army Research Office

ATTN: Dr. William Clark

P. O. Box 12211

Research Triangle Park, NC 27709-2211

Executive Summary

This is the final summary report of a project on graphene performed at Carnegie Mellon University from September 1, 2009 to August 31, 2012. The objectives of the project were twofold: (i) construction of a growth system for synthesizing high quality, large area graphene, and (ii) investigation of the properties of graphene for potential infrared sensing. The first objective has been successfully met; the second has yielded results that have been informative on future fabrication of graphene-based infrared sensors. We have determined, for example, that it is possible to dope graphene p-type or n-type with metals of appropriate work functions relative to it. Doping is essential for formation of p-n junctions that can be used in light detection. Furthermore, our results suggest that it may be advantageous to embed graphene in an optical cavity in order to enhance its absorbance so that a robust photocurrent can be generated when the device structure is illuminated. This requirement is in view of the single atomic- or bi-layer nature of graphene samples that are likely to be used in active media of detector structures.

1.0 Statement of the Problem Studied

Mechanically exfoliated graphene (usually in small areas) has been experimentally shown to produce the best analog transistors made from this material. The twofold objective of this project was to construct a growth system for synthesizing high quality, large area graphene, and to investigate its properties for potential infrared sensing.

1.1 Introduction

It is implicit that one of the key enablers for investigation of graphene for infrared sensing is a reproducible process for synthesizing large area films. The most widely used approach to producing the highest quality graphene, as already alluded to in the problem statement, is mechanical exfoliation; graphene made in this manner has yielded the best electrical properties to date. The major disadvantage of this approach is that only small pieces of graphene, typically on the order of several tens to about a hundred square microns of material can be obtained this way. Such small size samples are difficult to work with in the fabrication of uniform sets of devices that can be characterized in detail for a better understand their function. This would especially be true for single-pixel infrared detectors where the goal is to make focal plane imaging arrays out of the individual devices. The other approach to producing graphene is sublimation growth. Here, a silicon carbide substrate is heated in vacuum until the top silicon atoms leave the substrate by sublimation. What is typically left behind is a layer of carbon atoms on top of the silicon carbide substrate, bonded in a hexagonal crystalline form; this is graphene. In most practical situations, it is extremely rare that a monolayer of carbon (graphene) is left on the surface. More often than not, a rough structured surface comprised of regions of single layer and multiple layer graphene is obtained following sublimation. It is apparently difficult to control how many layers of graphene result from the sublimation process. One other issue about this approach is the use of silicon carbide substrate; it is inconvenient, expensive, and not compatible with CMOS technology for future scaling.

The most promising approach involves chemical vapor deposition using a carbon source on various metal substrates on which graphene is formed. After growth, the graphene layer(s) can be transferred to other more suitable substrates such as silicon or other any desirable substrate.
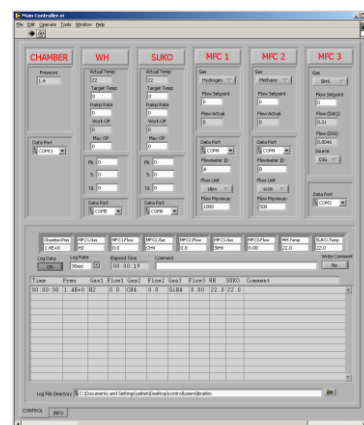
2.0 Summary of Key Results

2.1 Synthesis of Graphene

During the course of this work, we developed a unique method for synthesizing graphene that is a hybrid of the vacuum sublimation approach and the chemical vapor deposition scheme. Our technique anticipates future investigations of superlattices comprised of alternating layers of graphene and silicon carbide formed on silicon substrates. We describe the basic design of the

growth system in the following. The system is comprised of a cylindrical water-cooled metal vessel that can be evacuated to ultra-high vacuum pressures by a combination of mechanical and turbo-molecular pumps. The system is equipped with a 2-inch substrate heater that is surrounded by a water-cooling jacket, and capable of heating substrates up to 1500°C. Several sources have been installed in the chamber. For carbon, we have a solid carbon source and a methane (CH_4) gas source. In addition to these primary sources, we have also installed a facility for provision silane (SiH_4) gas. Three

Fig. 1: Photograph of a growth system constructed for synthesis of graphene.

process gases are also available in the system; these are nitrogen for purging the system, hydrogen for cleaning substrates, and argon as a carrier for silane and methane. We have instrumented the system with an *in-situ* reflection high-energy electron diffraction (RHEED) analysis tool; this tool will be used to analyze the evolution of surface structure in cases where a crystalline substrate is used for the growth of the graphene. This growth mode is akin to the molecular beam epitaxial process where a solid carbon is used as a source for carbon atoms for the synthesis of graphene.
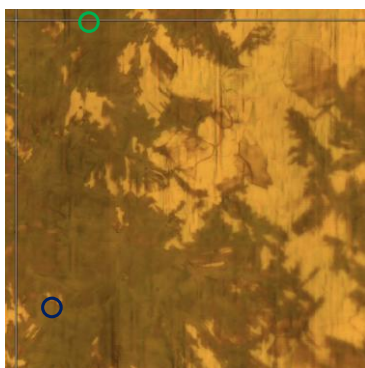

Fig. 2(a): An optical surface image of a nickel substrate with graphene grown on it.

An image of the growth system described in the foregoing is shown in Fig. 1. The sources described are installed from the top of the vessel as shown in the image. A sample loading port and the RHEED gun are clearly identifiable in the lower section of the photograph. The system is designed with automatic actuators for controlling the flow rates of gases; in addition, it has a shutter for controlling the molecular flux from the solid source. Control signals from these devices are interfaced to a computer that enables programmable operation. Shown next to the image of the vessel is a computer process sheet that displays the parameters during a typical process run.

Even though the growth system is designed to operate in two possible modes, the preliminary effort in this work was focused on using the system in the chemical vapor deposition mode. The results discussed in this report are based on the chemical vapor deposition mode of operation. In this mode, we use metal foils as substrates on which graphene is formed. Metallic

substrates such as nickel or copper facilitate growth of graphene via catalytic process. The basic process flow is as follows. The growth chamber is first pumped down to a base pressure of less than 10 milliTorr. For in-situ substrate cleaning step the substrate temperature is ramped at a rate of 20 to 30°C per minute to the ultimate cleaning temperature of 1050°C. At the substrate temperature of 700°C, hydrogen gas is turned on at the flow rate of 50sccm. The chamber background pressure is adjusted to about 0.1 Torr by carefully controlling the opening/closing of the gate valve between the chamber and the pumps. When the sample temperature reaches the desired value of between 900°C to 1050°C, the hydrogen flow rate is reduced to 25sccm, and methane gas is introduced into the system at the flow of 300sccm. The effective ambient chamber pressure for growth is eventually stabilized at 2.8 Torr. Graphene growth lasts for 10 to 20 minutes. Growth is essentially terminated when the hydrogen gas is progressively turned off at 100sccm. The methane flow rate is progressively reduced as the substrate temperature is ramped down at the rate of 20 to 35°C/min. When the substrate temperature is down to 700°C, the methane gas is turned off. Argon gas may be flowed through the system at this time. At a substrate temperature of 50°C, the chamber may be vented and the sample removed from the heater.

The essential chemical reaction that takes place during the synthesis of graphene is catalysis. When methane hits the surface of the heated metal substrate, it is cracked into carbon and hydrogen. The hydrogen is pumped away, while the carbon is absorbed by the metal. By cooling the substrate to room temperature at a controlled rate in the presence initially of hydrogen and then argon gas, the carbon floats to the surface and forms the hexagonally linked graphene structure. Depending on the type of metal used (copper or nickel in our case), actual growth temperature, and cooling rate, single or multiple layers of graphene can be formed on the surface of the metal.

2.2 Post Growth Characterization of Graphene

Following growth, one usually visually inspects the sample to determine if there is a deposit of graphene on it. This qualitative assessment should be followed up by a measurement of the Raman spectrum of the sample. We use a Renishaw Raman system to characterize the samples in our work. The spectra are measured with the graphene still on the metal substrates. For graphene on copper, care is taken to filter out the strong visible luminescence of the metal. Figure 2(a) is an optical image of a sample of graphene on nickel. The coverage of the
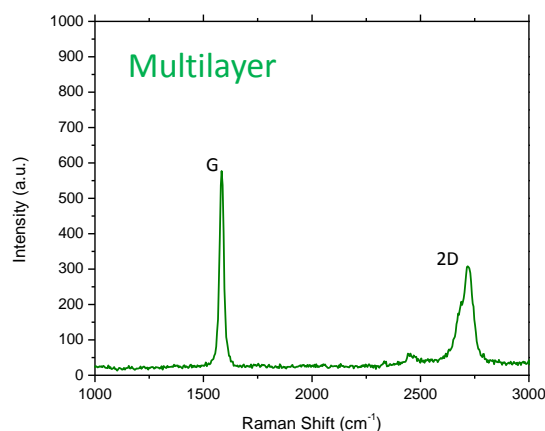

Fig. 2(b): The Raman spectrum of a multi-layer graphene region indicated by a green circle in Fig. 2(a).
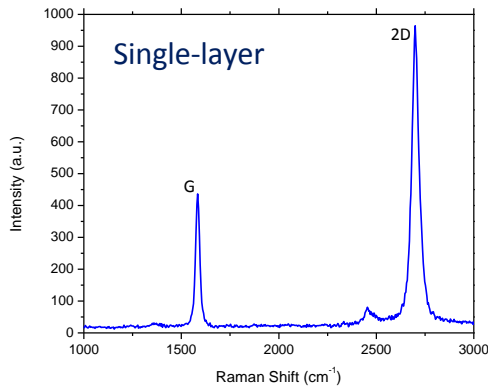
Fig. 2(c): Raman spectrum of the single-layer region indicated with the color blue in Fig. 2(a).

substrate is not complete. The darker regions of the sample are covered with graphene; the lighter colored areas are devoid of graphene. Raman spectra were measured in the regions identified in green and blue. Figure 2(b) shows the typical Raman spectrum measured from the green region of the sample where multiple layers of graphene are present. The spectrum shows the usual bands expected from carbon-based materials in general, and graphene in particular [1]. Two prominent bands labeled G and 2D at, respectively, $\sim 1580\,\text{cm}^{-1}$ and $\sim 2700\,\text{cm}^{-1}$ are clearly labeled. The G band is traditionally ascribed to zone-center optical phonons, and the 2D band is an overtone or harmonic (of $1350\,\text{cm}^{-1}$) that is present due to coupling of transverse optical phonons at the edge of the Brillouin zone. There is usually a defect-induced band identified as D at $\sim 1350\,\text{cm}^{-1}$; this is missing in the spectrum of Fig. 2(b), which is probably an indication that the particular region of the sample characterized did not have any defects. Notice, however, that the width of the 2D band is broader than that of the G band, indicating the multiple-layer nature of the graphene in this region. The Raman spectrum from the blue region is shown in Fig. 2(c). There is a suggestion of a bump at $\sim 1350\,\text{cm}^{-1}$, slightly to the left of the G band, and another definite bump to the left of the very prominent 2D band. The ratio of intensity of the 2D band to the G band is larger than two, indicating that single-layer graphene is found in this region of the sample. The spectral width of the 2D is also narrow, another indication of the single-layer nature of the material in this region. Both the spectral width and intensity of the 2D band are usually used as qualitative measures of the quality of graphene.

The coverage of the graphene over the metal surface depends on several growth parameters. These include gas flow rates, substrate temperature during growth, background chamber pressure, and the cooling rate of the substrate after growth. To illustrate the role that any one of these parameters plays, we have varied the substrate

(a)   (b)   (c)

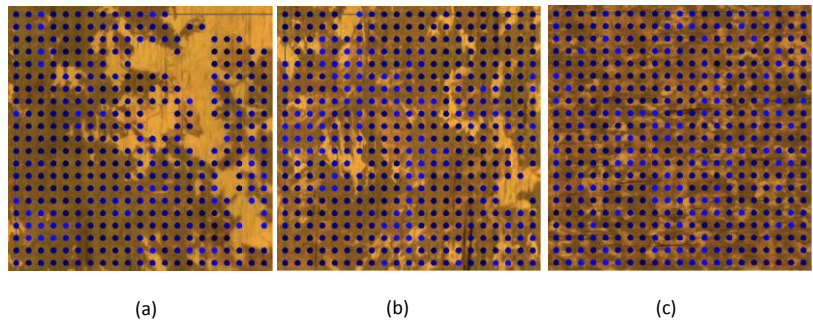**Fig. 3:** Optical images of the surface of nickel substrates on which graphene has been grown at the temperatures of (a) 1000$^{\text{o}}$C, (b) 950$^{\text{o}}$C, and (c) 900$^{\text{o}}$C.

temperature during growth on a nickel substrate. We show in Fig. 3, images of the sample

surfaces for graphene-on-nickel grown at three different temperatures. The blue dots represent locations on the sample surface where a Raman spectrum was measured. As before, the dark regions in the images are where graphene has been deposited and the lighter colored areas are there is no graphene. The image in Fig. 3(a) is for graphene synthesized at 1000°C. About 87% of the area of the substrate is covered with graphene, with 6% of that covered with a single or double layer graphene. The second image, Fig. 3(b), is of a substrate with graphene grown at 950°C; in this case the 97% of the substrate area is covered with graphene, with 13% of that covered with single or bilayer graphene. When the substrate temperature is lowered to 900°C, the image in Fig. 3(c) shows that the substrate is completely covered (100%) with graphene. The fraction of the area covered with single or bilayer graphene increases to 17%. What is clear from these growth runs is that the optimal growth temperature appears to be around 900°C; but still remains to be investigated are the parameters for 100% single-layer growth, and 100% bilayer growth. These are subjects for future work.

2.3 Potential of Graphene in Photodetection

Graphene is potentially attractive as an active medium for photodetection because it has a flat absorption spectrum due to its lack of a band gap. Furthermore, it has a very high relative absorbance compared to any semiconductor; a single atomic sheet of graphene, which is about 0.34 nm, absorbs 2.3% of any incident light. This is much higher than the absorbance of InGaAs—one of the most widely used semiconductors for near-infrared photodetection. It would require a 20-nm thick layer of $In_{0.53}Ga_{0.47}As$ to absorb an equivalent amount of light at 1.55μm [2]. On top of these, graphene has a very high electron mobility which is desirable for fast device operation. These advantages, however, are difficult to exploit because of two major challenges. First, a single sheet of graphene in a device with a nominal area of several hundreds of square microns is not large enough to generate sufficient signal current to overcome noise; second, it is not feasible to form conventional style p-n junctions in single- or few-layer graphene. A successful conception of a graphene phodetector is therefore likely to take a different form from that of a traditional semiconductor photodiode. In the following, we discuss our preliminary work aimed at exploring ways to make graphene-based photosensors.
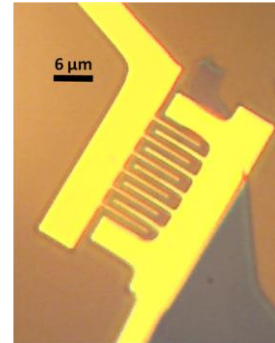

Fig. 4: Photomicrograph of a prototype metal-graphene-metal structure for investigating p-type and n-type doping in light sensing devices.

One approach to creating a p-n junction without doping in the traditional sense is to take advantage of the fact that graphene is a semimetal. At a junction between a semimetal and a metal, there usually is induced a galvanic effect. Therefore, a junction between graphene and a metal of appropriate work function should lead to a transfer of charge from one to the other, depending on the difference of the work functions. When in contact with a metal of a higher

work function, graphene donates electrons to the metal and is rendered deficient of electrons, making it p-doped. On the other hand, when it is in contact with a metal of a lower work function, electrons from the metal transfer to the graphene, making it electron-rich or n-type doped. This scheme, which was first theoretically discussed by Giovannetti *et al.* [3], for forming p- or n-type graphene was
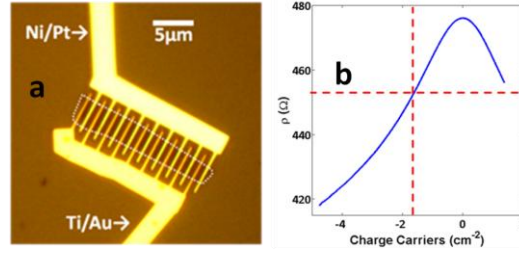

Fig. 5: (a) Comb-electrode structure of metal-graphene-metal device; (b) dependency of the resistivity on the areal carrier density (as extracted from measured I-V characteristics).

subsequently experimentally demonstrate by Mueller *et al.* [4]. We have fabricated devices with alternating metal contacts on the surface of the graphene. A typical such device is shown in Fig. 4. This device is comprised of graphene transferred onto the surface of a 300-nm layer of SiO_2 on top of a silicon substrate. The substrate is provisioned with a contact electrode that can be used to apply a field across the graphene through the substrate and the oxide layer. An electric field applied across graphene in this manner can be used to tune the carrier density in the layer, with the polarity of the field determining the type of carriers injected. The alternating electrodes are space 1 µm apart; each is 200 nm wide. The two metals out of which the alternating comb electrodes are made are Ti/Au (20/25 nm) and Ni/Pt (20/25 nm). The electrodes were defined by electron-beam lithography. The work function difference between the Ti and the Ni is about 0.8 eV; this translates into an electrical field across the 1-µm-gap between the electrodes of $8 \times 10^3 /cm$. The driving potential can be written as

$$\Delta V = \Phi_m - \Phi_G - \Delta E_{FG} \qquad (1)$$

where Φ_m is the metal work function, Φ_G is the work function of graphene and ΔE_{FG} is the change in the Fermi energy of graphene due to doping by application of a field between the substrate electrode and the one on top of graphene. The Fermi energy change in graphene is usually written as

$$\Delta E_{FG} = sign(n) \hbar v_F \sqrt{\pi n} \qquad (2)$$

where n is the dopant carrier concentration, $\hbar$ is Plank's constant and v_F is the Fermi carrier velocity. The carrier density, which controls the Fermi energy, can be tuned by the applied voltage field. From electrical transport measurements, one can see that it is related to the bias voltage through the relationship

$$n \equiv N - P = \varepsilon_{ox}(V - V_0)/t_{0x} \qquad (3)$$

where $N(P)$ is the electron (hole) concentration, ε_{ox} and t_{ox} are the silicon dioxide dielectric constant and thickness, V is the applied bias and V_0 is the charge neutrality (Dirac point) bias voltage.

To extract some of the parameters, the devices were characterized by current-voltage (I-V) measurements and by a scanning Kelvin probe. The measure data were fitted to a transport model for graphene so that the electron mobility and charge carrier density could be extracted. Shown in Fig. 5 is the dependency of resistivity (in the vertical axis) as a function of charge carrier density (in the horizontal axis, which should be multiplied by a factor of 10^{12}). The measurements were performed at room temperature and in air. The dashed red lines indicate the zero bias (intrinsic) doping condition for graphene samples in our experiments. The intrinsic
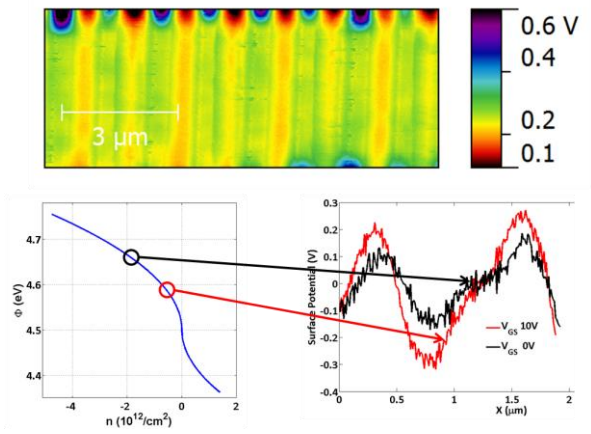

Fig. 6: Periodic variation of the surface potential (in color) due to the alternating n-type and p-type doping by the comb-like electrode metals on the device surface.

carrier denisty is calculated to be 1.5×10^{12} cm^{-2}. The Fermi energy at this doping level can be calculated from Equation (2). We can also evaluate the charge carrier mobility via $\mu = \sigma / ne$ to be $8650 \, \text{cm}^2 / \text{V-s}$.

To examine and verify the formation of p-n junctions between the alternating comb-like electrodes, we have used a scanning Kelvin probe system to measure the surface potential across the device. The potential profile of the different doping levels between the electrodes can be accessed through the conductive tip of an atomic force microscope as it passes over the sample for a given constant height separation between the tip and the surface of the device. The typical results of the Kelvin probe measurement are shown in Fig. 6. In this figure, the potential plot, given in the top of the figure as a color image, shows the alternating nature of the potential due to p-type and n-type doping. The data shown below the color image on the left is the direct Kelvin probe measurement of the work function over the sample as a function of carrier density. To the right of this plot is the measured Kelvin probe potential for no applied bias and for a 10-volt applied bias between the substrate and the electrode on top of the graphene as a function of lateral distance across the comb-like electrode structure. The two maxima in potential correspond to the two different electrodes comprised of Ti/Au and Ni/Pt. The alternating pattern of the potential profile clearly demonstrates that the alternating potential due to formation of the p-type and n-type regions between the electrodes is an indication of the success of the doping scheme.

2.4 Key Findings for Future Work

The results of this work have shown that large area graphene can be grown by the system designed and constructed during the project. Lessons learned from preliminary device structures

indicate that p-type and n-type doping—required for detector operation—can be achieved by deposition of metals with appropriate work functions relative to that of graphene. The major finding, however, is that despite the spectrally broad and relatively high absorbance of graphene (at 2.3%) compared to conventional semiconductors, the atomic-layer thickness of the material may be insufficient for absorption that would lead to a robust measurable photocurrent. Future work on phodetection will therefore explore the sandwiching of the graphene within a resonator. Such a structure would increase the number of times the light traverses graphene, thus leading to a compounded moderate absorption.

2.5 Publications from this Work

<u>Journal Papers</u>

1. A. Ramasubramaniam D. Naveh, and E. Towe, "Tunable Band Gaps in Bilayer Graphene−BN Heterostructures" Nano Letters, **11** 1070-1075 (2011).
2. A. Ramasubramaniam D. Naveh, and E. Towe, "Tunable band gaps in bilayer transition-metal dichalcogenides," Phys. Rev. B **84**, 205325-9 (2011).

<u>Conference Proceedings</u>

1. A. Ramasubramaniam D. Naveh, and E. Towe, "Tunable band gaps in bilayer graphene-BN heterostructures," Bulletin of the American Physical Society, APS March Meeting 2011 Vol 56(1) Paper ID: BAPS.2011.MAR.H37.5 (2011).
2. D. Naveh, A. Ramasubramaniam, and E. Towe "Electronic and transport properties of few-layer MoS_2 Crystals," Bulletin of the American Physical Society, APS March Meeting 2012 Vol. 57(1), Paper ID: BAPS.2012.MAR.Z17.1 (2012).
3. A. Ramasubramaniam D. Naveh, and E. Towe, "Tunable band gaps in transition metal dichalcogenides," Bulletin of the American Physical Society, APS March Meeting 2012 Vol. 57(1), Paper ID: BAPS.2012.MAR.V7.10 (2012).

<u>Technical Reports Submitted to ARO</u>
1. I. Kimukin, D. Naveh, E. Towe, "A Fundamental Investigation of Infrared Properties of Graphene for Potential Sensor Applications," September 1, 2010 – August 31, 2011.

2. I. Kimukin, D. Naveh, E. Towe, "A Fundamental Investigation of Infrared Properties of Graphene for Potential Sensor Applications," September 1, 2009 – August 31, 2010.

2.6 List of Participating Personnel Employed on the Project

(1) I. Kimukin, (2) D. Naveh, and (3) E. Szabo-Wexler

Bibliography

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